

Synthesis and Characteristics of β -Tri-Calcium Phosphate from Green Mussel Shell

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Abstract. The β -tri-calcium phosphate (β -TCP) is a calcium phosphate compound also called whitlockite. This form is more widely used because of its chemical stability, high mechanical strength and better bio-sorption when compared with α -Tri-calcium-phosphate. β -tri-calcium phosphate in this study was obtained from reactions of calcium oxide and phosphoric acid by precipitation method. Precipitation at 50 °C while sintering at 600, 700, 800, 900, and 1000 °C, in the sintering range of 1-5 hours. Green mussel shell has calcium carbonate (CaCO_3) content 60-70 % then calcined at 1000 °C to get calcium oxide (CaO). The result shows that β -tri-calcium phosphate optimum reached of 72% at sintering temperature 1000 °C for 5 hr sintering time. The crystal structure of β -tri-calcium phosphate formed is a rhombohedral crystal with a Ca / P ratio mole of 2.72

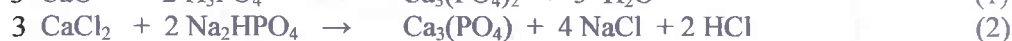
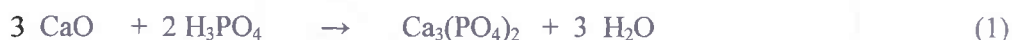
Key words: β tri-calcium phosphate, green mussel shell, sintering, precipitation.

1. Introduction

Calcium phosphate is the name given to a family of minerals containing calcium ions (Ca^{2+}) together with orthophosphates (PO_4^{3-}), meta-phosphates or pyrophosphates ($\text{P}_2\text{O}_7^{4-}$). Calcium can be obtained from various sources, such as natural mineral calcium carbonate and derived from organic sources such as eggshells, or other seafood shells. Calcium phosphate was prepared from phosphoric acid and oyster shells [1] and other composite as its function and characteristic development. [2-7]. Calcium phosphate (Ca/P) have chemical, biological, and mechanical properties similar to mineral phase of bone, and ability to bond to the host tissue, although it can disperse from where it is applied [2,4]. Calcium Phosphate ceramics have been widely used in tissue engineering due to their excellent biocompatibility and biodegradability. Ultimately, they are capable of replacing damaged bone with new tissue [5]. The application of calcium phosphate ceramics has become relatively to far and wide in the biomedical materials field, due to their biocompatibility with human hard tissue. Pure calcium hydroxyapatite (HA, $(\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2)$) and β -tricalcium phosphate (β -TCP, $\text{Ca}_3(\text{PO}_4)_2$) bioceramic powders, that have been synthesized via chemical precipitation techniques [6]. Nanocomposites based on of β -tri calcium phosphate (β -TCP) and 2.5-10 wt% merwinite nanoparticles were prepared and sintered at 1100-1300°C[8]

Various factors that affect the production of calcium phosphate include: degree of acidity (pH), combustion temperature (sintering), raw material composition, and the source of calcium is organic or inorganic. The Ca/P calcium phosphate usually in the range of 1.63 to 1.67

The calcium phosphate prepared by following reaction :



The purpose of this study was to produce the β -tricalcium phosphate from green mussle shell and to study the characterization by X-ray Diffraction (XRD) and prepared by heating an equimolar mixture of CaHPO_4 and CaCO_3 at 1500 C for 24 hours in Pt foil.

2. Materials and Method

This research is divided into two processes, the first process is the calcium production from green mussel shell and the second process is the calcium phosphate production. There are two process variables: temperature sintering (600, 700, 800, 900 and 1000 C) and time sintering (1, 2, 3, 4, and 5) minutes. The Block diagram of the research as shown in Figure 1.

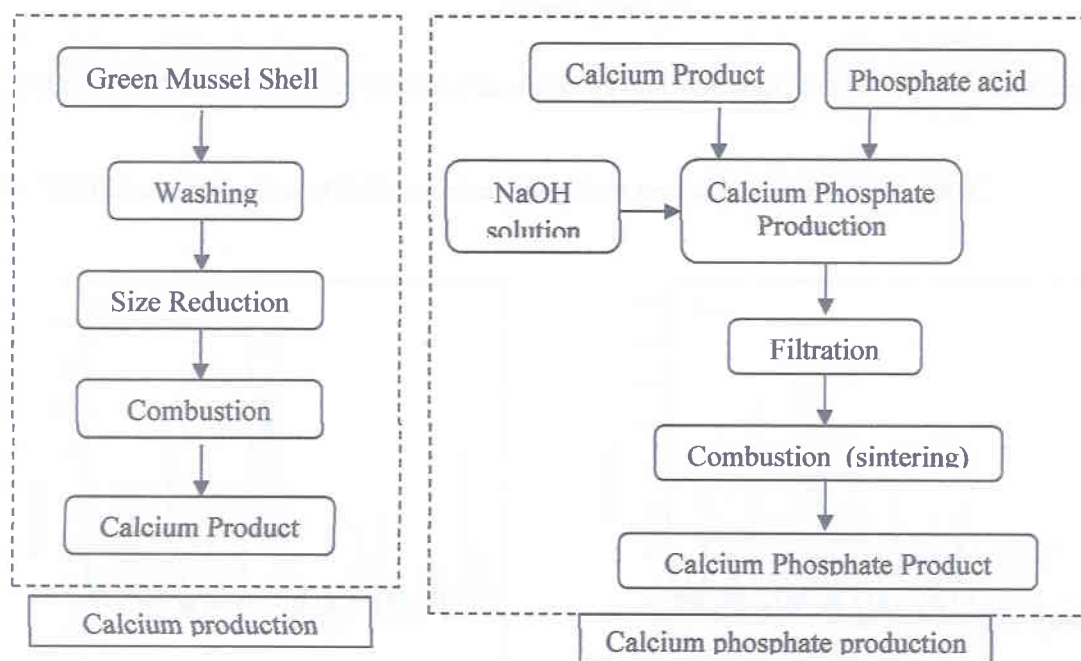


Figure 1. Schematic Diagram of Research

3. Result and Discussions

a. The effect of time and sintering temperature on Ca/P ratio calcium phosphate product

Figure 1 showed the effect of time and sintering temperature on Ca/P ratio of calcium phosphate product. The Ca / P ratio tends to decrease with increasing sintering time from 1 to 5 h. A sharp decrease in Ca / P from 4 to 3.14 occurs in an increase in sintering time from 1 to 5 h at a temperature of 800 °C. The increasing of sintering temperature from 600 to 1000 °C tend to decrease the Ca/P ratio except for temperature rise from 700 to 800 °C, Ca / P increases but then Ca / P decrease again until reached temperature of 1000 °C. The lower Ca/P obtained in sintering temperature 1000 C in the range of 3.04 to 2.72 for 1 to 5 h.

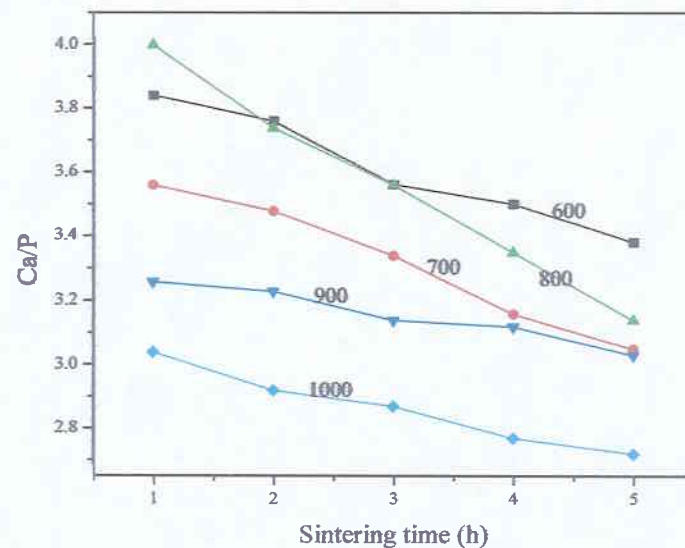


Figure. 1. Effect of time and sintering temperature on Ca/P ratio of calcium phosphate product

b. The XRD characterization of β -Tri-calcium phosphate product at temperature 600 °C

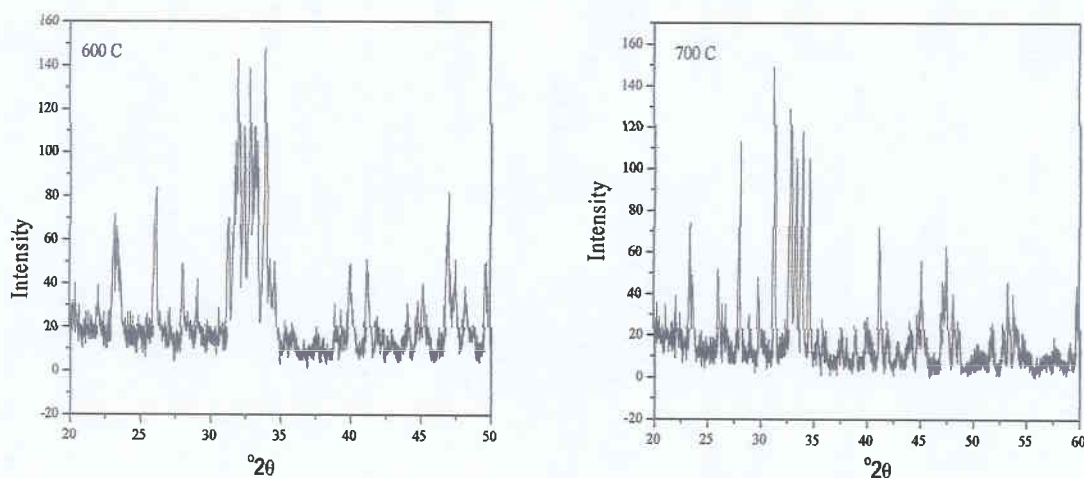


Figure. 2. XRD pattern for TCP product at temperature a) 600 °C and b) 700 °C

Figure 2 showed XRD pattern for TCP product at temperature 600 and 700 °C. There are two phases that occurred: Tri-calcium Phosphate (TCP) and Calcium oxide Phosphate (COP). The result found the pattern (Figure 3) as COP and CP, then obtained about 90% of TCP and 10% of COP at 600 °C and about 82% of TCP and 12% of COP. No β -TCP appeared in both samples β tri-calcium phosphate.

c. The XRD characterization of β -Tri-calcium phosphate product at temperature 800 °C

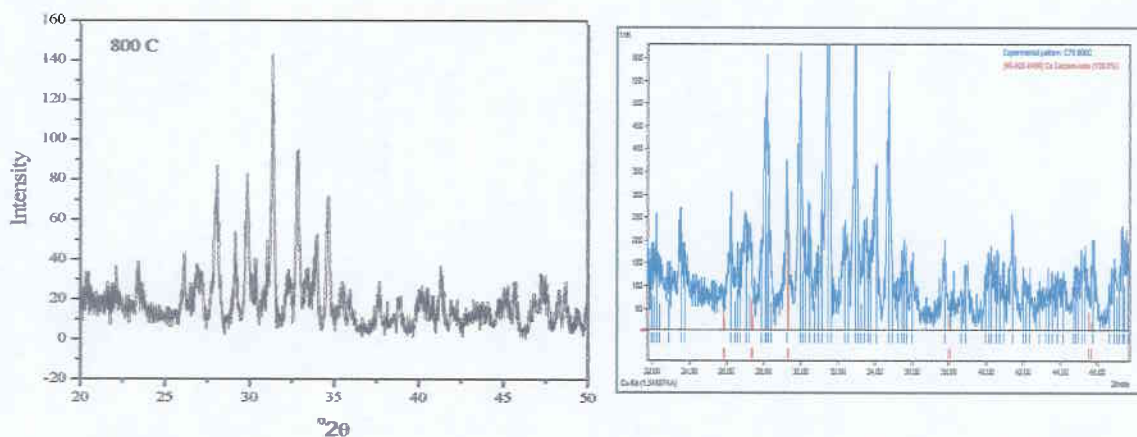


Figure. 4. The XRD pattern of TCP product at 800 °C

Figure 4 showed XRD pattern of TCP product at 800 °C. at the sintering temperature 800 °C for 5 h begin to form β Tri-calcium Phosphate. β tricalcium phosphate (TCP) characterized by the red line, which has 2 highest peaks at 2θ angles of 27.25° and 29.27°.

d. The XRD characterization of β -Tri-calcium phosphate product at temperature 900 °C

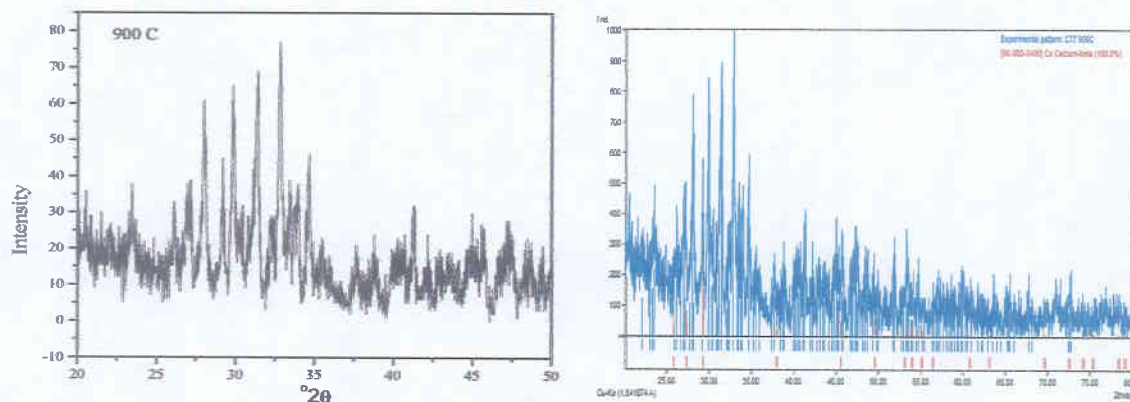


Figure. 5. XRD pattern of TCP product at 900 °C

Figure 5 showed the XRD pattern of TCP product at sintering temperature 900 °C. There are two phases that occur are Tricalcium Phosphate (TCP) and Calcium oxide Phosphate (COP). TCP value of 76% and COP 24%. In this sample phase β -TCP there are 3 highest peaks at 2θ angles of 25.95°, 27.22° and 29.24°.

e. The XRD characterization of β -Tri-calcium phosphate product at temperature 1000 °C

Figure 6 shows the XRD pattern of β -TCP product at sintering temperature of 1000 °C.. There are two phases that occur are TCP and COP. TCP value of 72% and COP 28%. Phase β -TCP appears characterized by four peaks at 2θ angle of 27.41, 29.37, 37.92 and 45.51°.

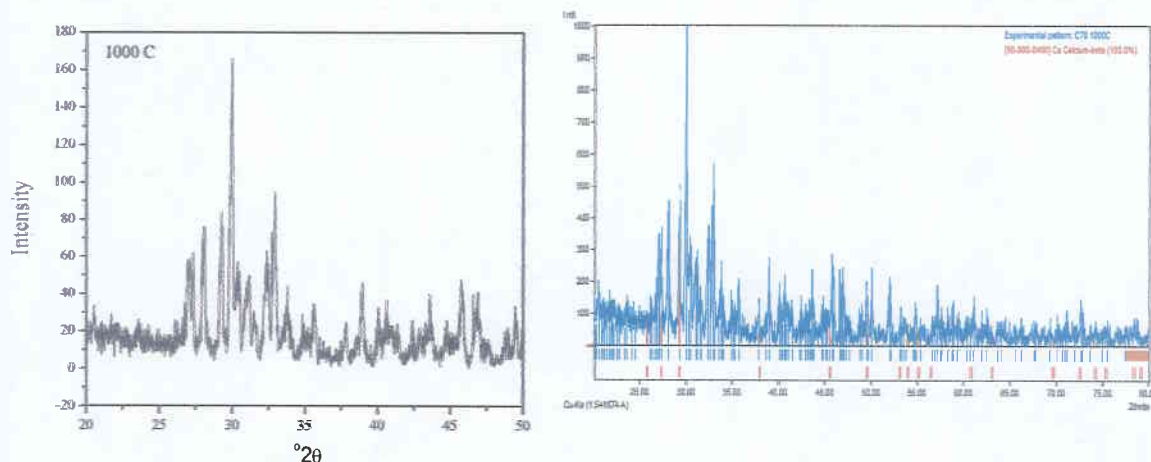


Figure. 6. The XRD pattern of β tri-calcium phosphate product

Figure 6 shows the XRD pattern of β -TCP product at sintering temperature of 1000 °C.. There are two phases that occur are TCP and COP. TCP value of 72% and COP 28%. Phase β -TCP appears characterized by four peaks at 2θ angle of 27.41, 29.37, 37.92 and 45.51°.

4 Conclusions

β -TriCalcium Phosphate from green mussel shell with Ca / P = 2.72 can be obtained at sintering temperature of 1000°C for 5 h, content element is Ca 74,28% and P 21,26%, Phase β -TCP appears characterized by four peaks at 27,41, 29,37, 37.92 and 45.51 angle °2 θ . The β -TCP crystal form indicates the rhombohedral.

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